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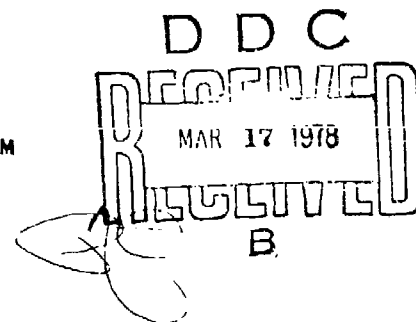
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CONTRACTOR REPORT ARSCD-CR-77002

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PILOT PLANT NITROCELLULOSE RECOVERY USING
DICHLOROMETHANE AS THE EXTRACTING SOLVENT
FOR BADGER ARMY AMMUNITION PLANT

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DECEMBER 1977



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
FIRE CONTROL
AND SMALL WEAPONS SYSTEMS LABORATORY
DOVER, NEW JERSEY

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
Contract Report ARSCD CR 77002		
4. TITLE (and Subtitle)		5. TYPE OF REPORT & PERIOD COVERED
PILOT PLANT NITROCELLULOSE RECOVERY USING DICHLOROMETHANE AS THE EXTRACTING SOLVENT FOR BADGER ARMY AMMUNITION PLANT.		Final Report Sep 69 - Nov 76
6. AUTHOR(s)		7. PERFORMING ORG. REPORT NUMBER
E. J. White, Olin Corp. J. M. Goldman, Technical Coordinator, ARRADCOM		
8. CONTRACT OR GRANT NUMBER(s)		
9. PERFORMING ORGANIZATION NAME AND ADDRESS		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
Olin Corporation Smokeless Powder Operations St. Marks, FL 32355		
11. CONTROLLING OFFICE NAME AND ADDRESS		12. REPORT DATE
ARRADCOM SCWSL Ammunition Division (BRDAR SCA 2F) Dover, NJ 07801		December 1977
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES
		72
		15. SECURITY CLASS. (of this report)
		Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)		
Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
Badger AAP, Baraboo, WI, monitored the execution of this contract. Funding was provided by AMSAR PPI C, Rock Island, IL 61201.		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Solvent extraction Nitrocellulose Propellant processing Dichloromethane Nitrocellulose recovery Ball propellant Solvent recovery Small arms		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
A Pilot Plant procedure was established for extracting BAAP FNI with methylene chloride using a three-stage, three hour 55°C extraction procedure with a 4.0:1.0 solvent-to powder ratio scaled up to 1000 gallon still size. Residual modifiers left in the FNI were reduced to the 0.4% level. The critical impact of size of FNI particles to be extracted upon the overall requirement was illustrated. Blends of ball propellants meeting WC846 and WC870 specifications using the methylene chloride extracted FNI were prepared.		

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20. ABSTRACT (Continued)

in order to test the effects of this extraction solvent on the finished powder. No traces of the methylene chloride extraction solvent could be found in powders as processed, and it may be safely assumed that the use of this solvent will not interfere with gun functioning or ballistic performance.

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INTRODUCTION

Work reported by Frankford Arsenal in FA-TR-75064, Alternate Solvents For Benzene In Nitrocellulose Recovery (August 1975), and in FA-TR-76049, Hazards and Risk Analysis Of Deterrent Extraction Process With Alternate Solvent (December 1975), indicated that methylene chloride could serve as a replacement solvent for benzene in the removal of deterrents from FNH in the BAAP extraction process.

The major objectives of the present program conducted at Olin-St. Marks were to scale-up Frankford Arsenal's laboratory findings to Pilot Plant equipment; define the operating conditions for a BAAP production unit; and prepare 100 lb each of WC 870 and WC 846 type propellants.

This report details the experimental work conducted at St. Marks to accomplish the objectives above.

EXPERIMENTAL DISCUSSION

1. FNH Extraction Work Plan

A "Test Program for Pilot Plant NC Recovery using Dichloromethane as the Extracting Solvent", (Appendix A), was supplied by Frankford Arsenal as a guideline for setting up a Pilot Plant extraction procedure. A work plan established for the extraction and solvent stripping work, (Appendix B), was set up using the test program as a starting point.

The extraction portion of the work plan was designed to test countercurrent extraction procedures at 35°C. in 10, 100 and 1000 gallon vessels. The 10 gallon vessel was to be used to determine the solvent to propellant ratio, the number of stages required, and the duration of each stage. Once a procedure for reducing the contaminants in the propellant to less than 1.0 weight percent had been established, a few extractions in a 100 gallon vessel were to be conducted both to confirm the results of the 10 gallon extraction and to evaluate the reuse of solvent. One final extraction in the 1000 gallon vessel was planned in order to confirm the tests in the 100 gallon vessel, to evaluate the process in near-production size equipment, and to produce sufficient material for processing into finished propellant.

Following the final stage of each countercurrent extraction, the solvent was to be stripped from the FNH and the FNH analyzed to determine the residual modifier and solvent levels. Target levels were 1.0% or less for the residual modifiers and about 0.5% for residual solvent in the extracted FNH.

The flow pattern for such extractions carried out in three stages is shown in Figure I.

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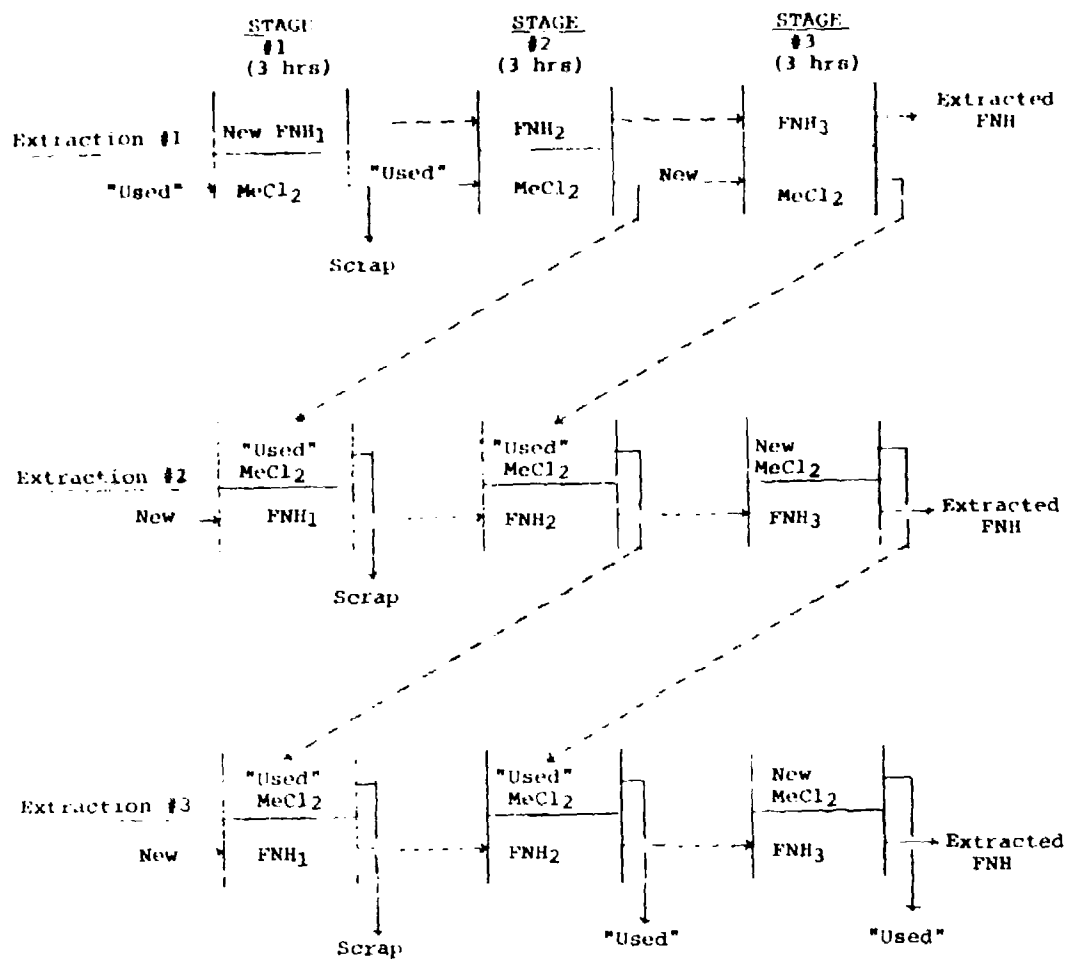


FIGURE 1
COUNTERCURRENT EXTRACTION PROCEDURE FOR THREE STAGE EXTRACTION

11. FNH Extraction And Stripping Data

Below are discussed the extractions conducted. A summary of all of the extractions, including procedure, and residual modifier and solvent content is shown in Table 1.

Table 1. Summary of Extractions @ 35°C.

Extraction Number	Number Of Stages	Length Of Stage (hrs.)	Solvent to Pdr. Ratio	Extraction Size (lbs.)	Final % Modifier	Final % R.S.
1	2	2	3.5:1.0	15	2.18	2.23
2	2	2	4.0:1.0	12	1.98	6.67
3	2	3	4.0:1.0	12	1.38	5.85
4	3	3	4.0:1.0	12	0.19	4.11
5	3	3	4.0:1.0	12	0.39	0.79
6	3	3	4.0:1.0	100	0.44	2.91
7	3	3	4.0:1.0	100	0.50	2.54
8	3	3	4.0:1.0	100	0.41	0.46
9	3	3	4.0:1.0	1,000	0.53	0.34

The first ten gallon still extraction was run using a solvent to powder ratio of 3.5:1.0 with two - two hour stages. Total residuals were at the 2% level as shown in Table 2. (All FNH analyses are adjusted to a dry basis. Where a residual solvent analysis was run, it is shown. Where no analysis was reported, 20% is assumed for simplified calculation.) Temperature charts of the actual extraction

Table 2. Extraction Data - Run No. 1
Two - 2 Hour Stages - 3.5:1.0 Solvent:Powder Ratio

Stage	Weight %			
	DNT	DPA	DBP	MeCl2
A. FNH				
Feed Stock	7.71	0.37	5.22	
After 1st Extraction	3.48	0.10	1.43	18.67
After 2nd Extraction	0.93	0.01	0.35	20.11
After Solvent Stripping	1.84	0.04	0.30	2.23
	1.27	0.04	0.64	2.93
B. Methylene Chloride				
Feed - 2nd Extraction	0.0	0.0	0.0	
Discharge - 2nd Extraction	0.56	0.02	0.08	
Feed - 1st Extraction	0.44	0.06	0.16	
Discharge - 1st Extraction	2.19	0.12	0.33	
After Solvent Stripping	1.11	0.02	0.20	

and solvent stripping are included for reference for this and all subsequent batches in Appendix C.

The second extraction was run exactly like the first except the solvent to powder ratio was increased to 4.0:1.0. The results of the experiment are shown in Table 3. The modifier content of the extracted FNH remained over 2% there being no difference, within experimental error limits, from the 3.5:1.0 extraction.

Table 3. Extraction Data - Run No. 2
Two - 2 Hour Stages - 4.0:1.0 Solvent:Powder Ratio

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	11.03	0.43	3.47	
After 1st Extraction	4.70	0.09	0.925	(20.0)
After 2nd Extraction	1.03	0.01	0.225	(20.0)
After Solvent Stripping	1.64	0.02	0.32	6.67
	1.81	0.02	0.41	6.48
B. Methylene Chloride				
Feed - 2nd Extraction	0.0	0.0	0.0	
Discharge - 2nd Extraction	0.35	0.02	0.04	
Feed - 1st Extraction	0.36	0.04	0.05	
Discharge - 1st Extraction	1.58	0.11	0.27	
After Solvent Stripping	0.02	0.06	0.03	

Analysis of the extracted FNH, obtained from the first two extractions, indicates that the ground FNH was not being extracted as rapidly as predicted by Frankford Arsenal's tests. St. Marks' test procedures were discussed with BAAP and Frankford Arsenal and judged to be within their specifications.

The difference in extraction rates could be attributed to a difference in granulation of the standard FNH's. A screen analysis of the ground FNH, provided by BAAP, revealed the existence of a large range in grain size, with 41.45% of the grains being over 0.079" diameter (see Table 4). Extracted FNH samples from the first two extractions were screened on a 0.0937 screen, and reanalyzed for modifier content, with results as shown in Table 5. The lower residuals level in the screened samples shows the critical effect of the

Table 4. Screen Analysis of FNH

Screen Size (inches)	Wt. % FNH Retained on Screen		
	BAAP Sample	FA Sample	
.132	10.05		
.111	6.40	16.45	14.6
.0937	9.35	9.35	11.8
.0787	15.65	15.65	11.1
.0661	11.49	11.49	10.9
.0555	11.61	11.61	10.4
.0465	10.68		
.0394	5.45		
.0331	6.18	22.31	21.3
.0247	5.40		
.0197	3.88	9.28	17.7
.0165	-		
.0130	2.12		
.0090	.70		
Pan	<u>.54</u>	<u>3.36</u>	<u>2.2</u>
Total	99.50	99.50	100.0
AGD		.0716"	.0680"

Table 5. Extraction Data
Extracted FNH; screened on .0937" screen, and unscreened

Run No.	Solvent to Pdr. Ratio	Extracted FNH Screen Analysis	Wt. %		Wc. % DBP	Total Modifiers %
			DNT	DPA		
1	3.5:1.0	As Received	1.84	.04	.30	2.18
1	3.5:1.0	.0937"/fines	1.13	.03	.19	1.35
2	4.0:1.0	As Received	1.64	.02	.32	1.98
2	4.0:1.0	.0937"/fines	.81	.01	.15	.97

size of the particle to be extracted. This is totally consistent with theory and indicates that variations in screen analyses from sample to sample will allow a substantial margin of error in final results. This factor is the probable explanation of the differences in residuals values shown between the two final samples analyzed in the first two runs. While recognizing that the wide granulation spread of the base stock complicated both the extraction process and the data obtained therefrom, the decision was made to proceed with the stock as provided recognizing that final production procedures might be modified through a change in control of the grinding process and the size of ground FNH supplied for extraction.

The third extraction was made using standard BAAP FNH in a two stage - three hour extraction procedure, with a 4.0:1.0 solvent to powder ratio. Table 6 gives the extraction data for this run. Lengthening the extraction time from two to three hours for each stage reduced the final modifier content of the extracted FNH to under 1.4%. The extracted FNH was screened and retested for percent modifiers. The 0.0937"/fines contained 0.84% modifiers and the 0.0787"/fines contained 0.78% modifiers.

Table 6. Extraction Data - Run No. 3
Two - 3 Hour Stages - 4.0:1.0 Solvent:Powder Ratio

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	11.15	0.45	3.62	0.0
After 1st Extraction	2.26	0.20	0.525	(20.0)
After 2nd Extraction	1.32	0.02	0.25	18.66
After Solvent Stripping	1.15	0.01	0.22	5.85
.0937"	0.77	0.01	0.06	(5.0)
.0787"	0.67	0.01	0.10	(5.0)
B. Methylene Chloride				
Feed - 2nd Extraction	0.0	0.0	0.0	
Discharge - 2nd Extraction	0.36	0.02	0.05	
Feed - 1st Extraction	0.38	0.06	0.08	
Discharge - 1st Extraction	1.91	0.14	0.51	
After Solvent Stripping	0.02	0.03	-	

The fourth extraction was completed using a three stage - three hour extraction procedure with a 4:1 solvent to powder ratio. The data for this extraction is listed in Table 7. Although the residual modifier level was acceptable after three stages, this run could not, by itself, be used as the basis for proceeding to the 100 gallon vessel for the following reason. One half hour into the third stage, the air compressor shut down. The third stage could not be completed until this was repaired. As a result the powder soaked in methylene chloride for about three hours prior to completion of the third stage. The additional contact time with the solvent is believed to have reduced the residual modifiers to levels lower than those which would have been expected had the extraction been run as planned.

Table 7. Extraction Data - Run No. 4
Three - 3 Hr. Stages - 4.0:1.0 Solvent:Powder Ratio

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	11.19	0.53	3.06	
After 1st Extraction	2.92	0.06	0.43	14.05
After 2nd Extraction	1.29	0.01	0.21	19.55
After 3rd Extraction	0.23	0.025	0.05	20.64
After Solvent Stripping	0.18	0.01	-	4.11
B. Methylene Chloride				
Feed - 3rd Extraction	0.0	0.0	0.0	
Discharge - 3rd Extraction	0.12	0.02	0.06	
Feed - 2nd Extraction	0.0	0.0	0.0	
Discharge - 2nd Extraction	0.53	0.04	0.14	
Feed - 1st Extraction	0.63	0.11	0.31	
Discharge - 1st Extraction	2.51	0.20	0.97	

A final ten gallon still extraction was completed to obtain more representative data on the procedure used in Extraction 4. As shown in Table 8 a final modifier content of less than 0.5% in the extracted FNH was realized after the third extraction confirming that the three step process would bring residual well under the 1.0% maximum level specified. The decision was made at this point to proceed to scale up to the one hundred gallon still level, initially using three extractions but with the obvious option to change to two if the results warranted.

Table 8. Extraction Data - Run No. 5
Three - 3 Hour Stages - 4.0:1.0 Solvent:Powder Ratio

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	10.89	0.51	2.95	0.0
After 1st Extraction	3.80	0.09	0.725	(20.0)
After 2nd Extraction	0.65	0.01	0.11	(20.0)
After 3rd Extraction	0.175	0.01	0.05	(20.0)
After Solvent Stripping	0.29	0.01	0.09	0.79
B. Methylene Chloride				
Feed - 3rd Extraction	0.0	0.0	0.0	
Discharge - 3rd Extraction	0.11	0.01	0.03	
Feed - 2nd Extraction	0.0	0.0	0.0	
Discharge - 2nd Extraction	0.39	0.01	0.07	
Feed - 1st Extraction	0.38	0.06	0.08	
Discharge - 1st Extraction	2.29	0.05	0.44	
After Solvent Stripping	0.22	0.01	0.07	

Three runs were made in 100 gallon still equipment, extracting 100 lbs. of ground FNH in three 3 hour stages using the 4:1 solvent:FNH ratio. The results from these runs are summarized in Tables 9, 10 and 11. In all cases the total residuals after two extractions ran well above the 1.0% level and fell to 0.5% or less after the third extraction. During these runs the solvent was employed in countercurrent pattern, i.e., new solvent was used each time for the third extraction step but solvent from the previous third extraction was used for the subsequent second extraction and that from the second extraction in the next first extraction. On the limited scale of work carried out there were sufficient natural solvent losses so that it was necessary to augment the used solvents with new solvent plus added residuals in order to maintain the specified solvent:FNH levels. This introduced some problems but did not change the pattern of results.

Table 9. Extraction Data
Run No. 6

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	10.84	0.40	3.53	
After 1st Extraction	4.5	0.16	0.55	(20.0)
After 2nd Extraction	1.50	0.03	0.20	(20.0)
After 3rd Extraction	0.35	0.01	0.05	(20.0)
After Solvent Stripping*	0.36	0.01	0.07	2.91
*Corrected for 2.91% MeCl ₂				
B. Methylene Chloride				
Feed 3rd Extraction	0.00	0.00	0.00	
Discharge 3rd Extraction	0.43	0.015	0.11	
Feed 2nd Extraction	0.11	0.02	0.11	
Discharge 2nd Extraction	1.28	0.06	0.38	
Feed 1st Extraction	0.63	0.03	0.15	
Discharge 1st Extraction	2.81	0.17	1.00	
After Solvent Stripping	0.05	0.21	0.11	

Table 10. Extraction Data
Run No. 7

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	11.65	0.26	3.71	
After 1st Extraction	3.75	0.06	0.64	(20.0)
After 2nd Extraction	1.78	0.01	0.19	(20.0)
After 3rd Extraction	0.47	0.00	0.09	21.3
After Solvent Stripping	0.44		0.06	2.54
B. Methylene Chloride				
Feed 3rd Extraction	0.00	0.00	0.00	
Discharge 3rd Extraction	0.20	0.01	0.04	
Feed 2nd Extraction	0.12	0.01	0.00	
Discharge 2nd Extraction	1.28	0.06	0.38	
Feed 1st Extraction	0.62	0.04	0.19	
Discharge 1st Extraction	2.83	0.17	1.09	
After Solvent Stripping	0.04	0.08	0.02	

Table 11. Extraction Data
Run No. 8

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	9.91	0.37	3.37	
After 1st Extraction	4.09	0.075	0.06	(20.0)
After 2nd Extraction	1.11	0.01	0.15	(20.0)
After 3rd Extraction	0.34	-	0.05	23.79
After Solvent Stripping	0.29	0.02	0.10	0.46
B. Methylene Chloride				
Feed 3rd Extraction	0.00	0.00	0.00	
Discharge 3rd Extraction	0.20	0.01	0.04	
Feed 2nd Extraction	0.15	0.01	0.03	
Discharge 2nd Extraction	0.90	0.04	0.20	
Feed 1st Extraction	0.77	0.09	0.18	
Discharge 1st Extraction	N/A	0.18	1.21	
After Solvent Stripping	0.02	0.07	0.07	

Reference to the extraction batch data shows that there is a lack of balance in the analytical results. When the loss in residuals in the ground FNH is compared with the pick-up of residuals in the solvent, it would appear that either the former results are low or the latter are high. This is illustrated in Tables 12 and 13 where the changes

in DNT and DBP, respectively, are used to calculate the solvent:FNH ratio which would bring the two analyses into balance. In general these show that the solvent predicted is less than that actually used. The dependence of FNH results upon particle size has been previously commented upon, but this in itself does not account for the total problem.

Table 12. DNT Extraction Pattern

	Loss in FNH	Weight % Gain in MeCl ₂	Calculated Ratio MeCl ₂ /FNH
Run No. 6			
3rd Extraction	1.15	0.43	2.67
2nd Extraction	3.12	1.17	2.67
1st Extraction	6.22	2.18	2.85
Run No. 7			
3rd Extraction	1.31	0.20	6.55
2nd Extraction	1.97	1.16	1.70
1st Extraction	7.90	2.21	3.57
Run No. 8			
3rd Extraction	0.77	0.20	3.85
2nd Extraction	2.98	0.75	3.97
1st Extraction	5.82	-	-
Run No. 9			
3rd Extraction	0.50	0.17	2.94
2nd Extraction	3.66	0.46	7.96
1st Extraction	6.16	2.25	2.74

Table 13. DBP Extraction Pattern

	Loss in FNH	Weight % Gain in MeCl ₂	Calculated Ratio MeCl ₂ /FNH
Run No. 6			
3rd Extraction	0.15	0.11	1.36
2nd Extraction	0.35	0.27	1.30
1st Extraction	2.98	0.85	3.51
Run No. 7			
3rd Extraction	0.10	0.04	2.50
2nd Extraction	0.45	0.38	1.18
1st Extraction	2.07	0.90	2.30
Run No. 8			
3rd Extraction	0.10	0.04	2.50
2nd Extraction	-0.09	0.17	N/A
1st Extraction	3.31	1.03	2.31
Run No. 9			
3rd Extraction	0.05	0.05	1.00
2nd Extraction	0.77	0.18	4.28
1st Extraction	6.16	2.25	2.74

The conclusion is that the available data cannot be used quantitatively but that it is sufficient to support the extraction profile. Within the limits of the reliability of the data, small variations within the runs do not give critical changes in residuals.

One final run was made under these conditions moving up in scale to 1,000 lbs. of unextracted FNH in a one thousand gallon still. This is identified as Run No. 9 and the results are shown in Table 14. These follow the pattern previously established with one possible critical exception, the residuals content after the second extraction fall just under the 1.0% specification limit. This single result is not sufficient to justify recommendation of a two step process, but it does point up that reductions in the total overall extraction cycle may well be feasible in scale up to production and should be investigated in any final process establishment.

Table 14. Extraction Data
Run No. 9

Stage	Weight %			
	DNT	DPA	DBP	MeCl ₂
A. FNH				
Feed Stock	10.67	0.31	3.30	
After 1st Extraction	4.51	0.06	0.86	(20.0)
After 2nd Extraction	0.85	0.01	0.07	(20.0)
After 3rd Extraction	0.35	0.01	0.04	(20.0)
After Solvent Removal	0.46	-	0.075	0.34
B. Methylene Chloride				
Feed 3rd Extraction	0.00	0.00	0.00	
Discharge 3rd Extraction	0.17	0.01	0.05	
Feed 2nd Extraction	0.20	0.01	0.04	
Discharge 2nd Extraction	0.66	0.045	0.22	
Feed 1st Extraction	0.67	0.06	0.17	
Discharge 1st Extraction	2.92	0.18	1.12	

The above discussion has dealt with the process requirements for extraction of residual modifiers from the FNH in order to present a composition suitable as a nitrocellulose raw material for BALL POWDER manufacture. The process as described is incomplete until the extraction solvent itself has been removed and separated from the extraction material. During the period of this

study a specification of 0.5% maximum residual solvent was established for the finished material. Based on laboratory studies Frankford Arsenal recommended azeotropic distillation for removal of the bulk of the solvent with a final rapid heating to complete the solvent removal. Their work showed that solvent levels could be reduced by this method from about 6% to 0.5% in approximately nine hours.

The procedure described is best carried out in facilities wherein the amount of heat being supplied for solvent distillation is controlled on the basis of volume of vapors (or condensate) being produced. Unfortunately the still equipment available for this current study was not so equipped and efforts to hold at the 38-39 C. azeotrope temperature were generally inadequate. As a result, although residual solvent levels were reduced to the desired level, the process followed does not permit any definition of optimum distillation cycle temperature-time patterns. The results obtained are shown in Table 15.

Table 15. Solvent Stripping From FNH

Batch No. 6			Batch No. 7		
<u>Time (hrs)</u>	<u>Temp (°C)</u>	<u>Wt. %MeCl₂</u>	<u>Time (hrs)</u>	<u>Temp (°C)</u>	<u>Wt. %MeCl₂</u>
0	27	17.86	0	20	21.3
3 1/4	36	5.67	3	43	8.77
4 1/4	39	4.96	5	46	7.58
5 1/4	43	4.40	9	62	6.40
6 1/2	73	3.96	11	74	5.18
7 1/2	87	2.90	12	99	2.81
			13	99	2.54

Batch No. 8			Batch No. 9		
<u>Time (hrs)</u>	<u>Temp (°C)</u>	<u>Wt. %MeCl₂</u>	<u>Time (hrs)</u>	<u>Temp (°C)</u>	<u>Wt. %MeCl₂</u>
0	25	23.79	0	25	21.48
1 1/4	36	18.24	2 1/4	32	18.01
6	36	16.01	4 1/2	36	17.23
7	37	15.14	5 1/2	37	17.24
8	40	12.81	6 1/2	38	15.83
9	44	10.93	7 1/2	40	15.17
10 1/4	52	8.73	8 1/2	42	15.77
11	66	6.92	10 1/2	53	10.25
12	78	4.78	11 3/4	65	7.12
13	90	2.44	13	77	5.20
14	98	1.71	14	89	2.63
15 1/2	99	0.46	15	99	1.74
			16	99	1.50
			17	99	0.51
			18	99	0.39

Anticipating problems of control of heat input at the 38-39°C. level, Run No. 6 was made with a ten inch vacuum applied. Initial solvent removal proceeded rapidly, but the temperature rose above the azeotrope level well before the lower solvent levels were reached. This distillation was not carried to completion but it should not have deviated far from the nine hour 6% to 0.5% solvent levels predicted in the Frankford work. The vacuum procedure complicates solvent recovery so the next run, No. 7, was made under atmospheric pressure conditions. Again initial solvent levels dropped rapidly but temperatures rose rapidly and extended solvent removal times resulted. Run No. 8 was treated more cautiously with the result that temperatures initially remained too low for rapid solvent removal but even so did not hold at the desired level. The final thousand pound batch suffered from the same problem compounded by a faulty temperature control unit. However, the final product showed the desired low residual solvent level and times were not inconsistent with the rates of solvent removal reported from the laboratory.

Overall it was shown that the 0.5% solvent level can be achieved in the 15-17 hour range even with very limited controls. Under conditions of distillate control these times should be reduced. Such optimization must await implementation of the production scale operation.

As a footnote to the above work, it should be noted that a check was made for any possible breakdown of the solvent under repeated use. Solvent, as drained from the first stage extraction of Run No. 9, was refluxed in laboratory glassware for 24 hours. IR scans were made of the materials as new and after this treatment. These proved to be essentially identical indicating no breakdown of the solvent. This is shown in Figure II.

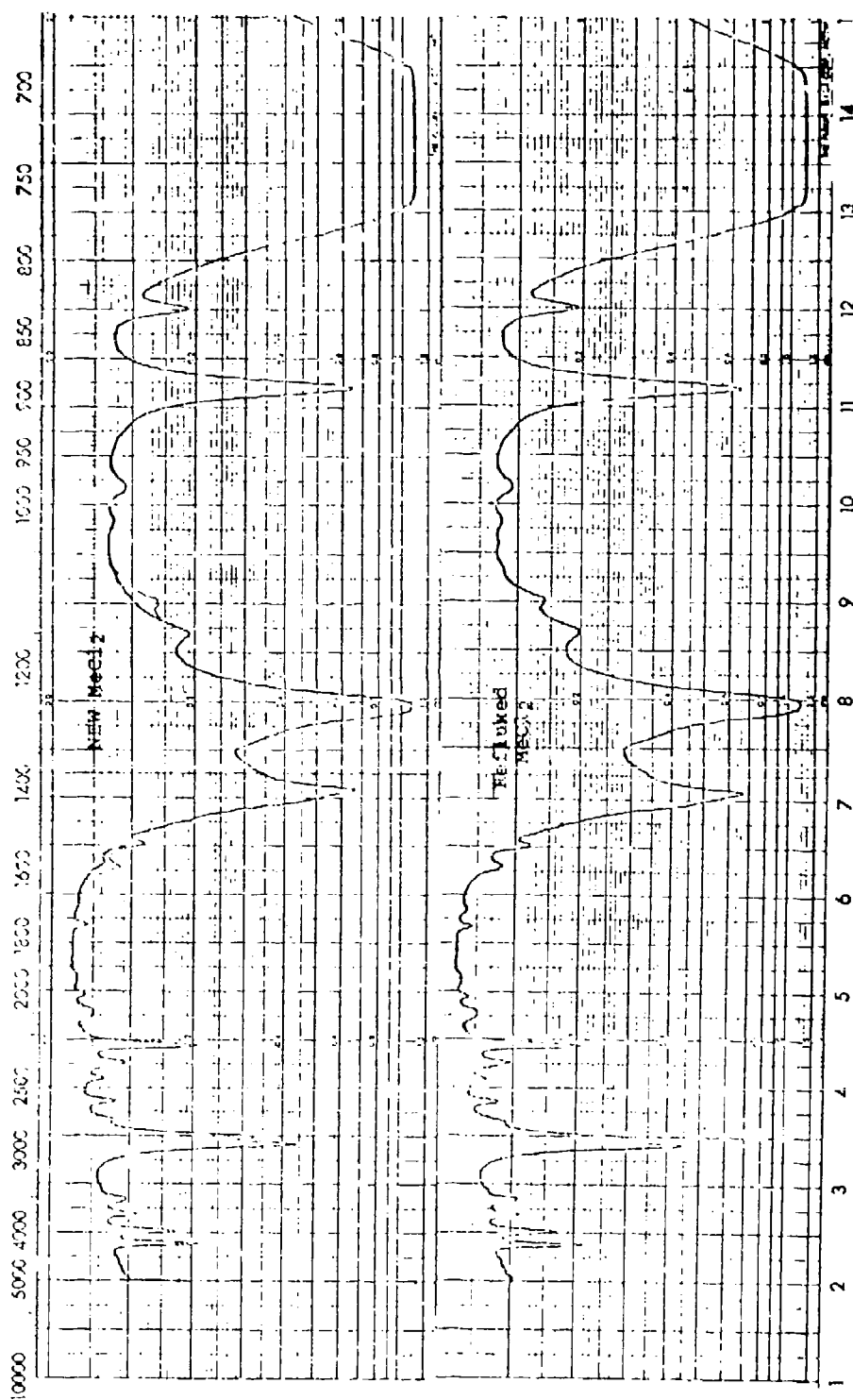


FIGURE 11

Wave-length (Microns) - Infra Red Spectrophotometer Comparison - New and Refluxed MeCl₂.

111. Hardening Data

The second phase of this study was to use the extracted FNH in the hardening, coating and finishing of propellant powders equivalent to the present WC 846 and WC 870 BALL POWDERS. In the planning of this work, it was recognized that one major problem would be to quickly arrive at a suitable process in the one thousand gallon pilot still to produce sufficient single base grains in the .034"/.028" size range as required for finishing to WC 870. This size grain is at the extreme range of the still's capabilities and the formula for its processing is sensitive to variations in the extracted FNH feed. Each hardening made was treated, therefore, as a problem in maximizing large diameter powder yields.

The initial extracted stock was enough to make three hardenings. The basic procedure for these are included in Appendix D, while summary data of the product are shown in Table 16 below. The first two runs, AG 3150 and AG 3151, yielded an undersized product with no significant amount of overs/.028". However, they did produce roughly 25-30% .028"/.020" stock as required for finishing to the WC 846 propellant. The third batch, AG 3152, was run at lower agitation levels in an effort to increase average grain size. While larger material was made, it proved to be of low specific gravity and was not acceptable for finishing by coating.

Table 16. Summary of Batch Hardenings

<u>Hardening Number</u>	<u>AG3150</u>	<u>AG3151</u>	<u>AG3152</u>	<u>AG3154</u>	<u>AG3156</u>
Batch Size (lbs.)	300	400	425	460	409
Solvent And (lbs.)	900	1,280	1,200	1,300	1,145
MeCl ₂ in used solvent %	0	0	0	0	0
MeCl ₂ in FNH %	0	0	0	0	0
.034"/.028" cut (lbs.)	15	15	scrap	70	100
G.D.	-	-	0.895	.930	.918
Sp.G.	-	-	1.479	1.550	1.518
AGD (inches)	-	-	N/A	0.0318	0.0287
.028"/.020" cut (lbs.)	100	100	-	-	120
G.D.	0.916	-	-	-	0.891
Sp.G.	1.563	-	-	-	1.538
AGD (inches)	0.0215	-	-	-	0.0247

Additional hardenings were made by reworking the off size and off specification materials from the first three batches. Some process modification was required for the handling of the rework, but sufficient WC 870 base grain material was produced in hardenings AG 3154 and AG 3156 so that coating studies could be initiated. The powder stocks produced varied in density so that two separate stocks were available; base powders produced through AG 3154 were of the design density and that from AG 3156 was lower than would have otherwise been used.

While the extracted FNI contained very small quantities of methylene chloride residues upon the completion of the extraction steps, the feed used in the hardening process no longer showed the presence of the solvent. The disappearance of methylene chloride from the extracted FNI is attributed to its being leached by process water. Therefore, any difficulties in obtaining base grain material having consistently acceptable density are attributable to process sensitivity not to the interaction of methylene chloride. The absence of residual methylene chloride in the final product is desirable since, if present, it would be a cause of gun barrel corrosion.

IV. Coating And Finishing Data

Seven coatings were made and then appropriately blended in order to produce acceptable WC 846 and WC 870 propellants. Coating studies were handicapped by the limited quantities of WC 846 and WC 870 base grain material available. The blending of the coated propellants to meet product specifications was difficult because portions of the coated material had low specific gravity and low nitroglycerine levels. Despite these problems, acceptable WC 846 and WC 870 propellants were produced, although in less quantity (< 100 lbs.) than planned. Relevant process and product data appear in Tables 17, 18, 19 and 20 and in Appendix F.

Table 17. Stocks Prepared For Blending To WC 846
Speed - Coating Summary

Base Powder	N.G. Impreg.	Det. Coating	Final Pdr.	Batch # lbs	NG Wt. %	DBP Wt. %	Web (in.)	G.D. g/ml
AG-3150/3151	N9202	D9937	AB20685-6	100	9.21	3.91	.0147 .0145	.926 .930
AG-3150/3151	N9214	D9943	AB20689-3	100			.014	
		D9947*	AB20669-3	68	3.52	5.70	.0137	.900
AG-3156	N9243	D10110	AB20712-9	3	8.99	5.65	.013	.952
			AB20712-10	2			.015	
			AB20713-1	2			.017	
			AB20721-5	47	8.99		.0128	
AG-3156	N10054	D10113	AB20717-2	2	7.42	6.22	.0112	.911
			AB20717-3	2			.015	
			AB20717-4	2			.0172	
			AB20721-4	60	7.42		.0168	

*D9947 is a re-coating of D9943.

Table 18. Stocks Prepared For Blending To WC 846
Speed - Test Firing In 7.62mm M80 Ball Ammunition

Powder	M&V Wt. %	Charge grains	Velocity fps	Pressure psi
AB20685-6	0.84	46.7	2711	52,700
AB20724-1	0.85		2741	55,700
AB20693-6	0.62	41.6	2622	51,300
AB20712-9		49.0	2485	34,600
AB20721-5		48.5	2322	27,400
AB20717-4		45.0	2741	50,300
AB20721-4		49.0fc	2504	33,100
AB20724-2	20 1/2 Pts AB20724.1	47.0	2728	50,800
	28 Pts. AB20721-4			
AB20728-5	20 1/2 Pts AB20724.1	48.0fc	2731	45,900
	28 Pts. AB20721-4	47.5	2749	48,100
AB20736-4	AB20728-8	47.0	2762	47,600

Table 19. Stocks Prepared For Blending To
WC 870 Speed - Coating Summary

Base Powder	N.G. Impreg	Det. Coating	Final Pdr.	Batch #	lbs	NG Wt. %	DBP Wt. %	KNO ₃ Wt. %	SnO ₂ Wt. %	G.D. (g/ml)
AG-3154	N9219	D9945	AB20693-5	70		9.35	5.20	0.66	0.84	.942
AG-3156	N9237	D10107	AB20706-8	65		4.98	6.36	0.97	1.00	.923
AG-3156	N9821	D9772	AB20717-10	25		9.05	6.15	0.54	0.68	.901

Table 20. Stocks Prepared For Blending To
WC 870 Speed - Test Firings In 20mm M56 Rounds

Powder	M&V Wt. %	Charge grains	Velocity fps	Pressure psi
AB20693-5	0.97	577	3374	59,900
AB20706-8	0.82	593fc*	3168	43,700
AB20717-10	1.01	600	3182	39,000
AB20723-8	50% AB20693-5	603	3343	52,200
	50% AB20717-10			
AB20728-6	85% AB20723-8	578	3363	55,900
	15% AB20693-5			
AB20732-3 (X3492)	AB20728-6	595	3380	54,000

*fc: full case

X-3491, the WC 846 equivalent, was prepared by conventional blending of two of the four coatings initially prepared. Note should be made that the fast component of this blend, N9202/D9937, was prepared from a suitably high density base stock and performs much as would be predicted from its web and deterrent level. Coating N9214/D9943 intended to be ballistically slow for blending with N9202/D9937 had too low a nitroglycerine level to be used in the preparation of finished propellant. The third and fourth coatings, N9243/D10110 and N10054/D10113, were made on lower density base stock and an effort was made to compensate for this by increasing the deterrent level, with some natural resulting loss in ballistic efficiency.

X3491, a thirty-four pound blend, was made up from components of coatings N9202/D9937 and N10054/D10113. The increase in pressure at low temperature with the X-3491 is within specification, but not desirable. This is attributed to the physical properties of that portion of the base powder

on the low side of the density requirement and consequent rolling aimed at increasing this. The result was that the minimum density specification on the blend was met, but the one fraction of the lot had a resultant tendency to crack, becoming brittle at -65°F. and partially fragmenting. With base stocks of proper density, the problem is routinely controlled with WC 846 and should be in any continued work here also.

Sufficient base powder was available for only three coatings for blending to WC 870 speed. The base stock for two of these was on the low density side. The procedure was again followed working to blend fast and slow components. The second of the three coatings showed up abnormally low in nitroglycerine level and was used in preparing a WC 870 blend. The first and third coatings did, however, furnish blend components and a fourteen pound blend, X3492, was finished.

No trace of chloride was found in either the coatings or final blends of WC 846 or WC 870.

For the record, samples of X3491 and X3492 are being placed in surveillance for proper record of long term chemical stability. The balance of these blends are available for any additional ballistic or chemical evaluation.

CONCLUSIONS

1. A three stage countercurrent extraction technique, using methylene chloride as the extraction solvent, effectively reduced the level of unwanted materials from the FNH to less than 0.50% by weight. This exceeds the specification of less than 1.00% by weight of residual modifiers.
2. There is strong evidence that a two stage countercurrent extraction process is feasible with its attendant economies of operation.
3. The granulation of the ground FNH feed stock significantly affects the efficiency of extraction.
4. Although not optimized, solvent stripping tests have shown that the residual methylene chloride in the FNH can be reduced to less than 0.5% by weight.
5. Acceptable WC 846 and WC 870 propellants can be made from nitrocellulose recovered by methylene chloride extraction of FNH.
6. The use of methylene chloride in the extraction of FNH has no apparent effect on subsequent ball propellant operations.
7. Since no trace of methylene chloride was found in the finished propellants, no adverse effects on gun barrel wear would be expected from the use of this extraction solvent.

RECOMMENDATIONS

1. Establish reduced level of solvent to base powder ratio that will give a three step extraction process with a residual extractable level in the FNN of less than 1.0%.
2. Establish modified ground powder size range which will permit a two step extraction process with a residual extractable level in the FNN of less than 1.0%.
3. Establish on a production scale an optimum distillation procedure for reduction of residual methylene chloride to less than 0.5% in the extracted product.
4. Determine the means to implement the use of methylene chloride in the extraction process at BAAP.
5. Determine the cost of using methylene chloride in production operations at BAAP.

APPENDIX A

FRANKFORD ARSENAL TEST PROGRAM

APPENDIX A

FRANKFORD ARSENAL

Test Program for Pilot Plant NC Recovery Using Dichloromethane as the Extraction Solvent

I Extraction Phase

Two process variables are to be investigated as a result of bench scale studies. They are solvent-to-powder ratio and duration of each stage. The number of stages is not to be considered a variable. Two stages will be used in pilot plant extractions unless approval for variation from that is specifically received from Frankford Arsenal. The temperature for all extractions will be 35°C. The starting processing variable designations are as follows for the first extraction attempt: Solvent-to-powder ratio, 3.5:1.0; duration of each stage, 2 hrs. The solvent for the first stage in all cases will contain 0.00754 weight fraction of modifiers in the following ratio of DNT-to-DBP-to-DPA, 10:3:1. If the first two-stage extraction attempt does not yield propellant with <1.0 percent weight modifiers, the following strategy should be used to achieve acceptably extracted powder. The solvent-to-powder ratio can be raised to as high as 4.0:1.0. If this measure fails to lower the level of residual modifiers in the powder, then the duration of each stage can be raised in, at minimum, 15 minute increments to as much as 3 hours per stage. If these measures fail, then a meeting among Badger AAP, Olin-St. Marks, and Frankford Arsenal personnel will be held to review all data and decide upon an alternative plan of action.

1.1 Solvent Stripping Phase

After draining dichloromethane from the second extraction stage, add 2.70 weight units of water for every 1.00 of powder. Under agitation, raise the temperature slowly (~3°C per 15 minutes) until it reaches 38-39°C. This is the azeotropic temperature for dichloromethane and water. The temperature should level off naturally in this range. The heat input should be reduced to 259 Btu/lb. powder-hr. until the temperature rises to, at least, 45°C. At this point the heat input may be increased to raise the temperature to 99°C as rapidly as possible and then hold it there for 5 minutes. It is important to add heat slowly until safely past the azeotropic temperature, because it has been shown that this strip is a diffusion-controlled operation. Too high a heat input has

caused the azeotrope to be passed and the amount of solvent stripped and recovered greatly reduced. It might be interesting to vary the heat input somewhat to alter the azeotropic portion of the operation to see the extent of improved or reduced solvent removal in order to determine the optimal heat input.

The powder should be analyzed for residual dichloromethane. A method for this analysis has been developed and will be provided when needed. Samples can be taken throughout the strip for this purpose or, at the minimum, at the end of the operation. The amount of solvent removed should be correlated with the analysis for dichloromethane in the powder.

III Solvent Recovery Phase

It is the opinion of Frankford Arsenal engineers that batch distillation of dichloromethane in the pilot plant would serve little utility, since such data are available from bench scale testing. A continuous stripping column will be used in the full-scale recovery of dichloromethane; therefore, no additional information can be gained from pilot plant batch distillation recovery of dichloromethane.

APPENDIX B

WORK PLAN FOR EXTRACTING FNH WITH METHYLENE CHLORIDE

APPENDIX B

WORK PLAN FOR EXTRACTING FNH WITH METHYLENE CHLORIDE

A. Extractions

1. Initial two stage extractions will be made in ten gallon stills until correct solvent to powder ratios, and extraction time, are found (initial solvent to powder ratio - 3.5:1.0).
2. Solvent into the first stage of each batch will contain .00754 weight fraction modifiers in the ratio of: Dinitrotoluene to Dibutylphthalate to Diphenylamine, 10:3:1.
3. Solvent for the second stage of each batch will be pure methylene chloride in these extraction batches.
4. If the extracted FNH from the first extraction contains more than 1% modifiers go to #5, if not go to #8.
5. The second batch will be extracted exactly like the first, except that the solvent to powder ratio will be 4.0:1.0.
6. If the extracted FNH contains more than 1% extractables the length of extraction will be increased to three hours at the 4:1 ratio.
7. If the propellant made from the best extraction batch still has > 1.0 weight % modifiers, the number of stages will be increased to three.
8. If after any of the extraction batches, a powder with < 1.0 weight % modifiers can be made, then all further extractions will be made using that solvent to propellant ratio, duration of stage, and number of stages. The testing for correct solvent ratio and length of extraction will stop at this time (after it has been demonstrated that the correct parameters have been found).
9. After determining the extraction condition, work will begin on optimizing the solvent strip procedure.
10. After the solvent strip procedure has been identified, along with an extraction procedure, three 100 lb. countercurrent extractions will be completed.
11. No solvent will be recovered by batch distillation.
12. One more extraction will be made with 1,000 lbs. of FNH using the test procedure previously obtained and modified in the 100 lb. extraction.

B. Solvent Make-Up

1. For the first stage of the initial extraction, the modifiers added to methylene chloride are to be calculated as follows:

$$\frac{14X}{(\text{Solvent ratio}) (\text{Wt. of Powder}) + 14X} = .00754$$

where:

X = weight of diphenylamine
3X = weight of dibutylphthalate
10X = weight of dinitrotoluene

2. For the second stage of initial extraction, fresh solvent is to be used.
3. For subsequent extraction, corrections should be made in accordance with analytical results obtained from previous runs.
4. For the first stage of any initial three stage extraction, the modifiers in the solvent may be calculated as in 1. above.
5. For the second stage of the first three stage extraction, modifiers should be added in accordance with 3. above.
6. For the third stage of the first three stage extraction fresh solvent is to be used.
7. For any subsequent three stage extractions, corrections should be made in modifier contents in accordance with analytical results obtained.

(In cases 4, 6 and 7 actual solvents recovered from previous runs will be available for use as a major portion of the solvent required.)

8. The three 100 lb. extractions will be run so that they will follow a production countercurrent extraction procedure. The (used) solvent for the first extraction will be synthesized. However, the second extraction will be run with used solvent from the first extraction, and the third extraction will be run with used solvent from the second. This procedure will give us data on the accuracy of the original estimate of modifier content of the used solvents.

C. Solvent Stripping

1. Add 2.7 lb. of water per lb. of FNH (original extraction weight) in the still and add drained powder from second stage and agitate (at 50 RPM).
2. Raise temperature slowly (3°C. per 15 min.) until reaching 38°C. At this point the temperature will level off. Cut back the heat to 1°C./15 min. until the temperature reaches 45°C. then heat the still rapidly until the temperature reaches 99°C. (at maximum steam input). Hold the temperature at 99°C. for five minutes and then cool to ambient temperature. Solvent will be dumped into used (first stage) solvent barrel.
 - a. Solvent in solvent receiver will be analyzed for impurities.
 - b. Water will be drained from powder and dumped into sewer.
3. Samples of powder will be analyzed before, during and after stripping to determine residual solvent levels.
4. Sample final extracted powder for weight percent modifiers. If less than 1%, run a second batch under the same conditions to confirm results.
5. Stability of powder will be determined after stripping.
 - a. Store extracted powder under water.

APPENDIX C

EXTRACTION AND STRIPPING PROCEDURES TEMPERATURE TRACES

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C-15	Extraction No. 9.....	46
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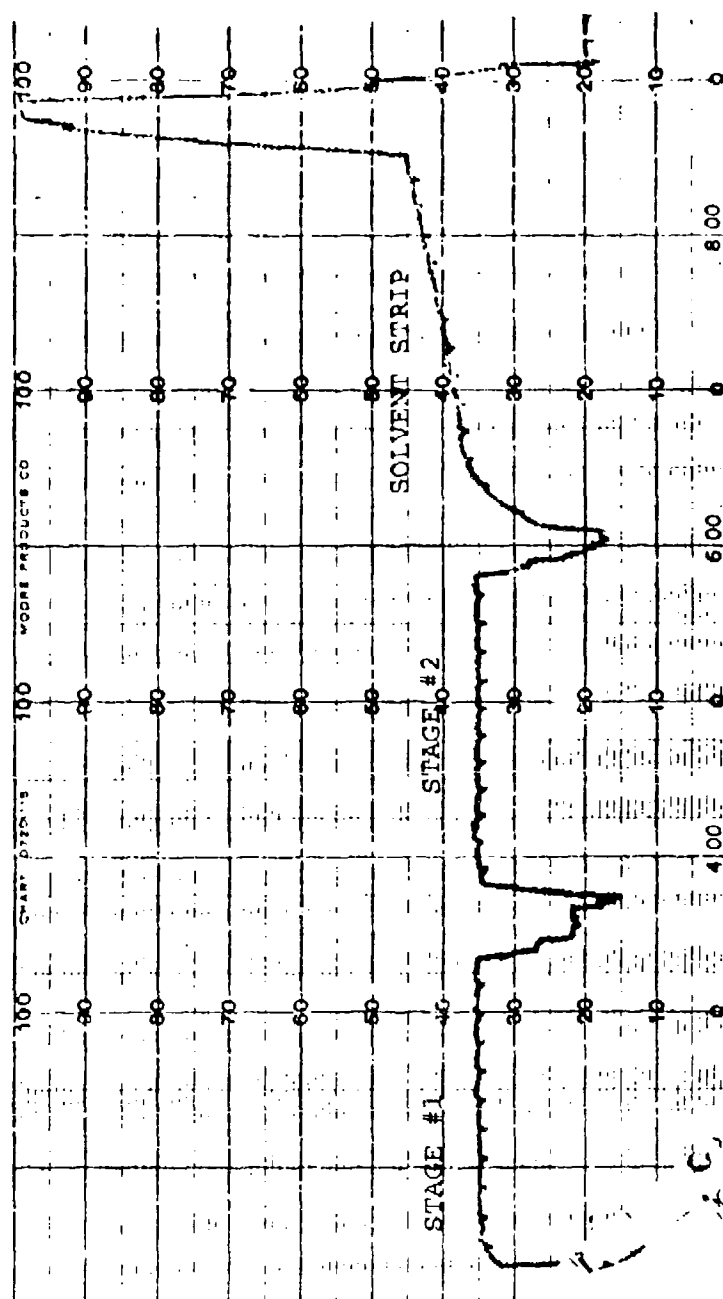


FIGURE C-2
Temperature trace for Extraction 2

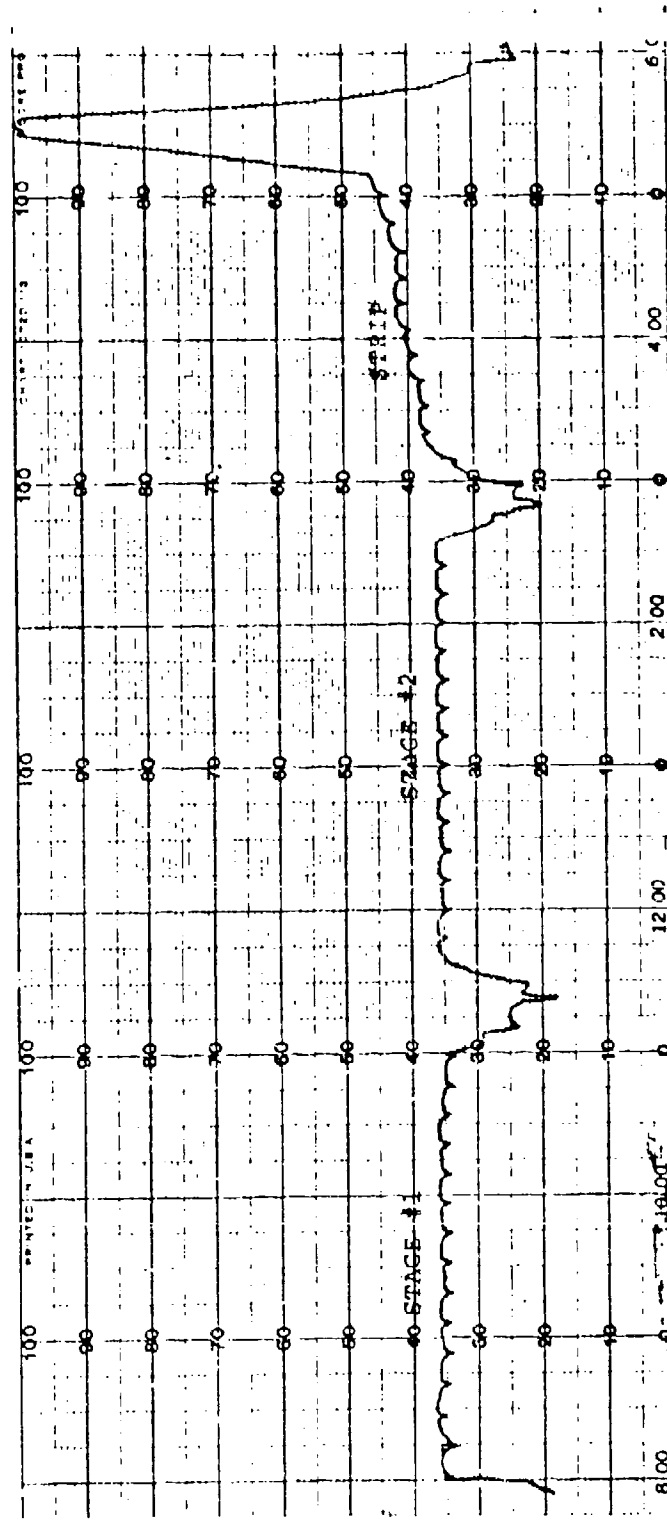


FIGURE C-3
Temperature trace for Extraction 3

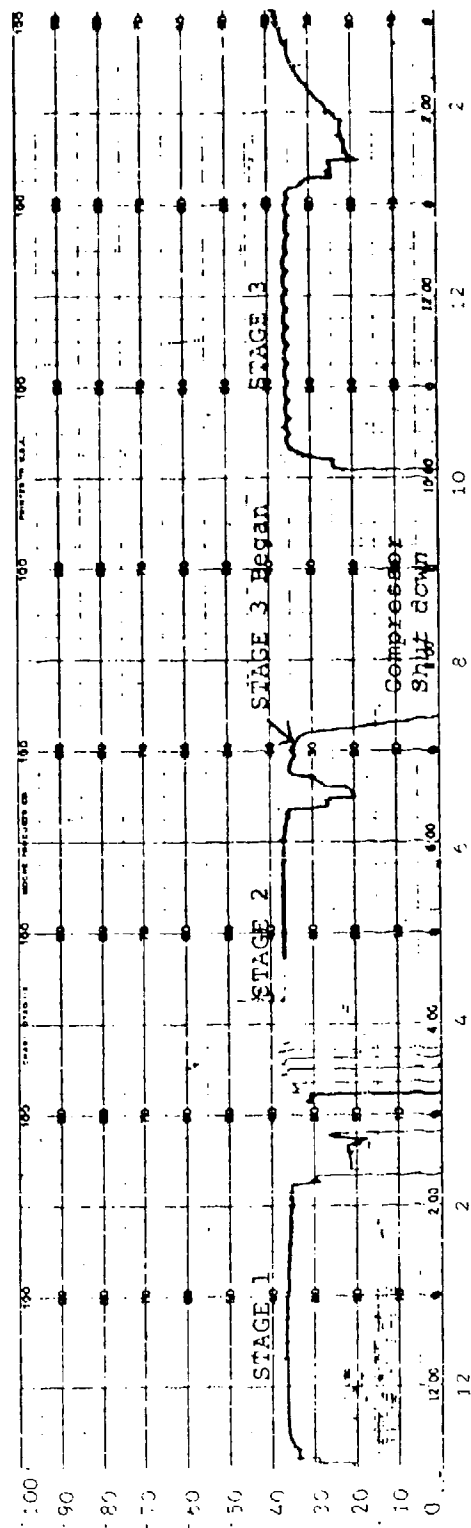


FIGURE C-4

Temperature trace for Extraction 4

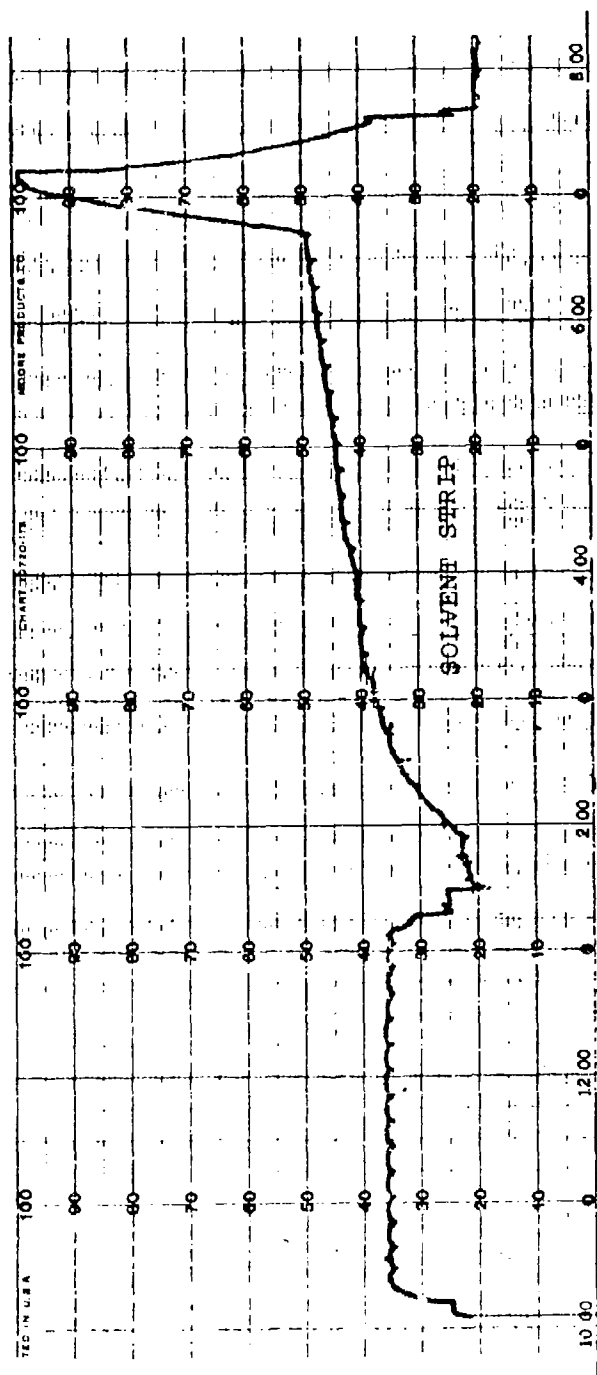


FIGURE C-5
Temperature trace for solvent strip - Extraction 4

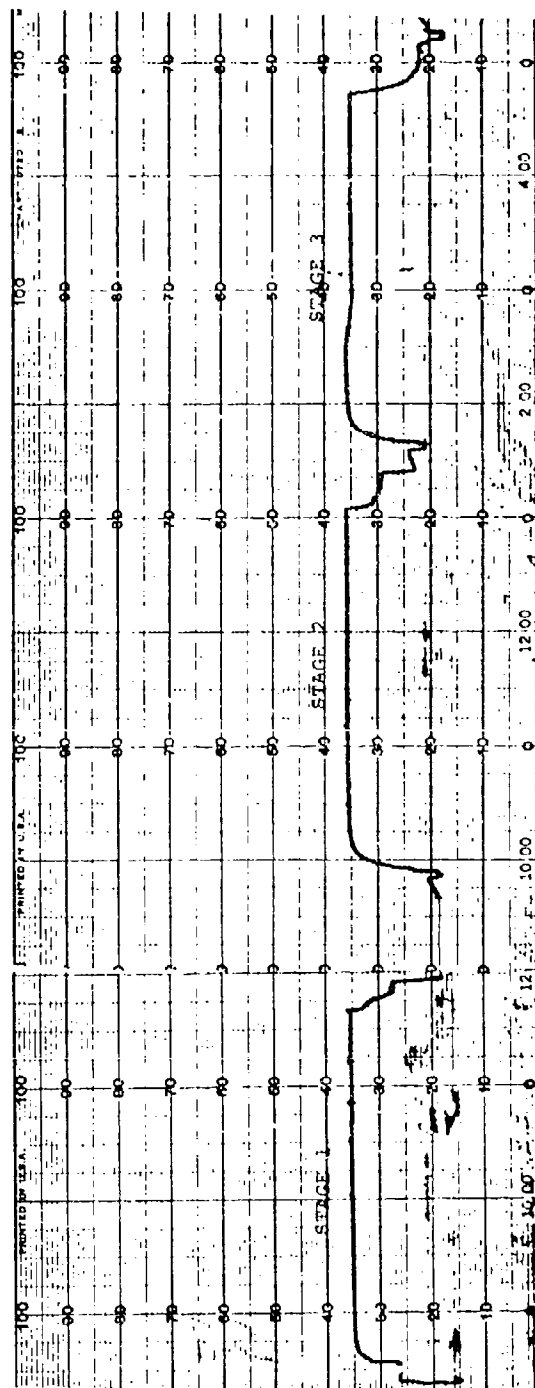


FIGURE C-6
Temperature trace for Extraction 5

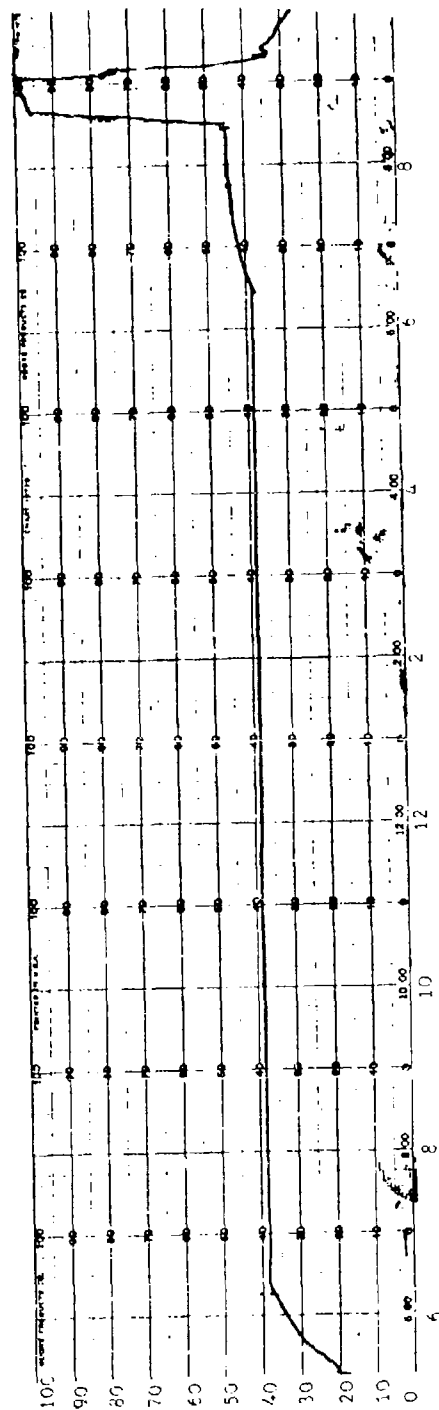


FIGURE C-7
Temperature trace for solvent strip - Extraction 5

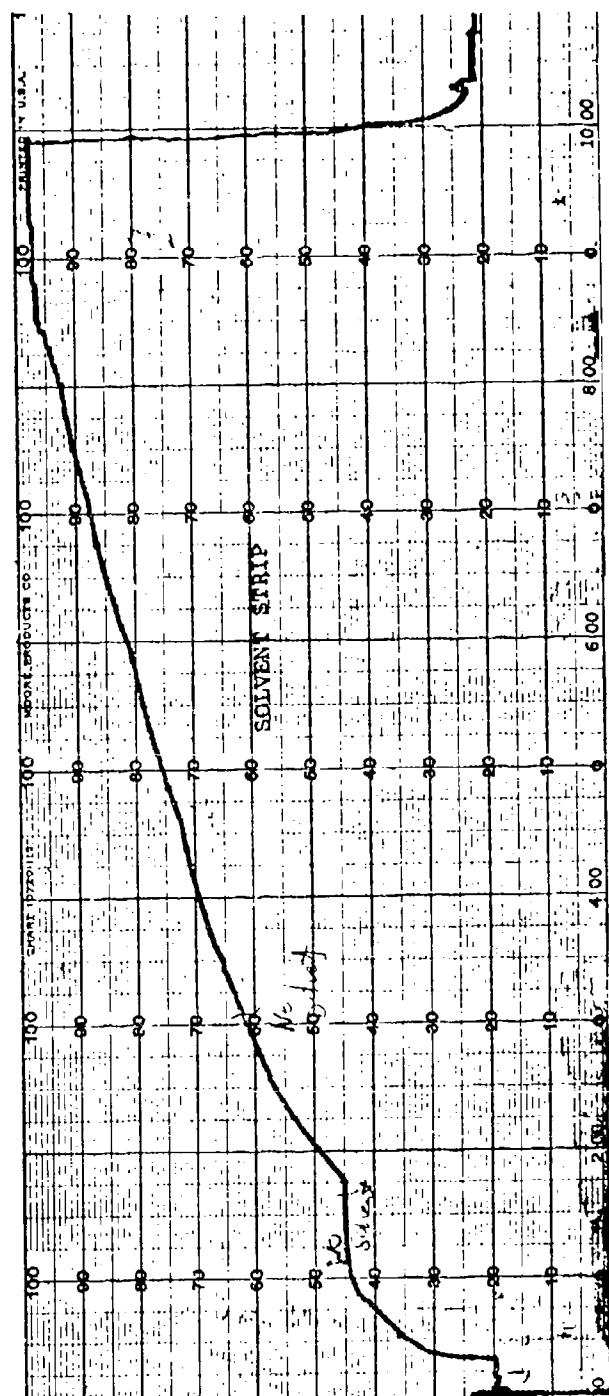


FIGURE C-8
Temperature trace for second solvent strip of FNH from Extraction 5

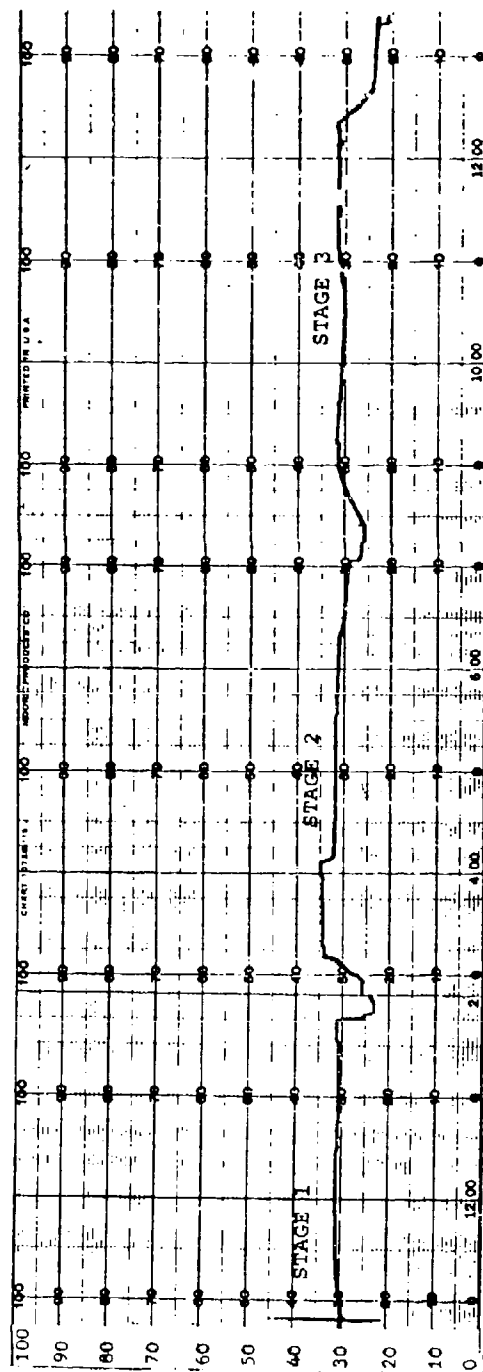


FIGURE C-9

Temperature trace for Extraction 6.

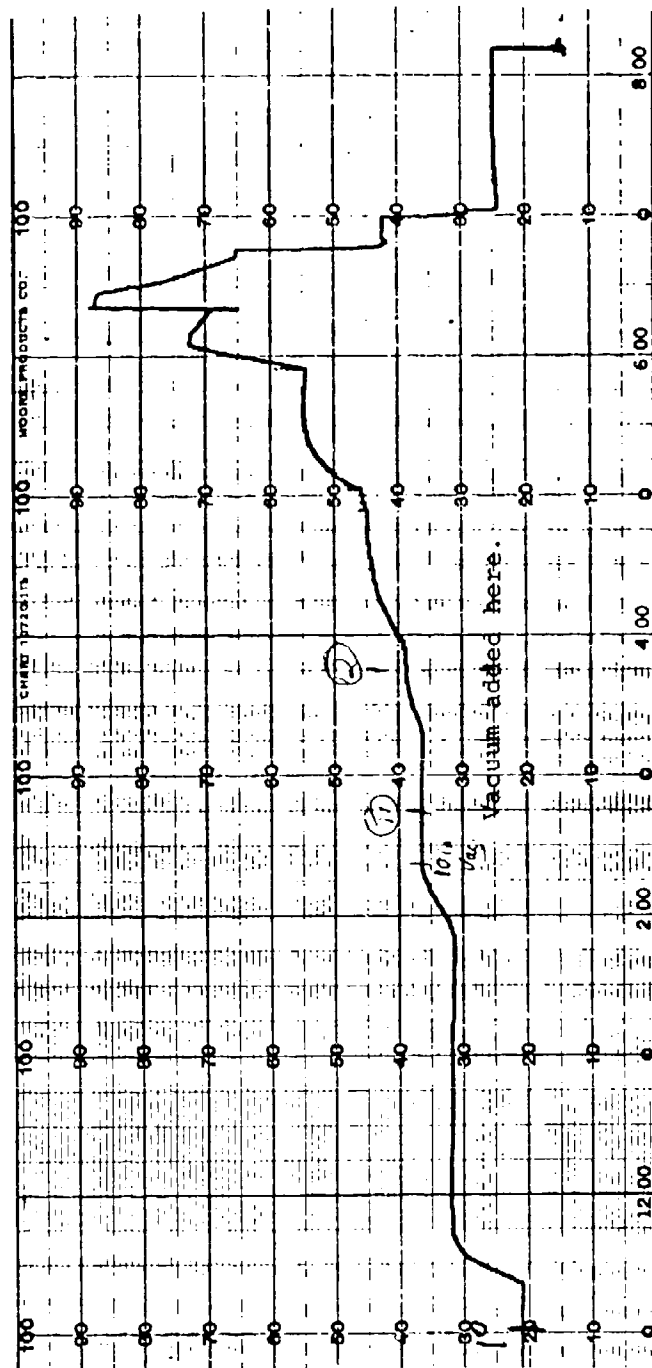


FIGURE C-10
Solvent strip for Extraction 6

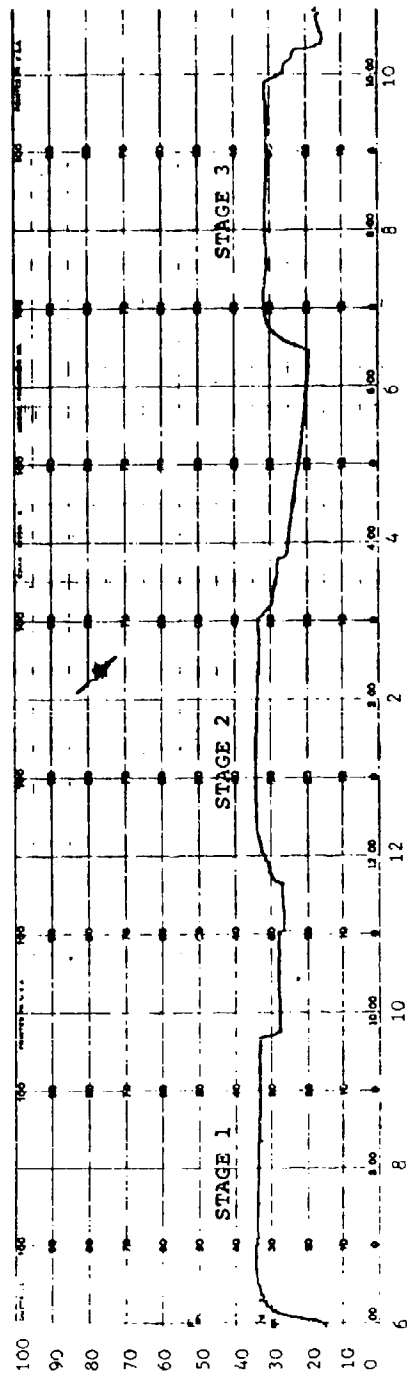


FIGURE C-11
Temperature trace for Extraction 7

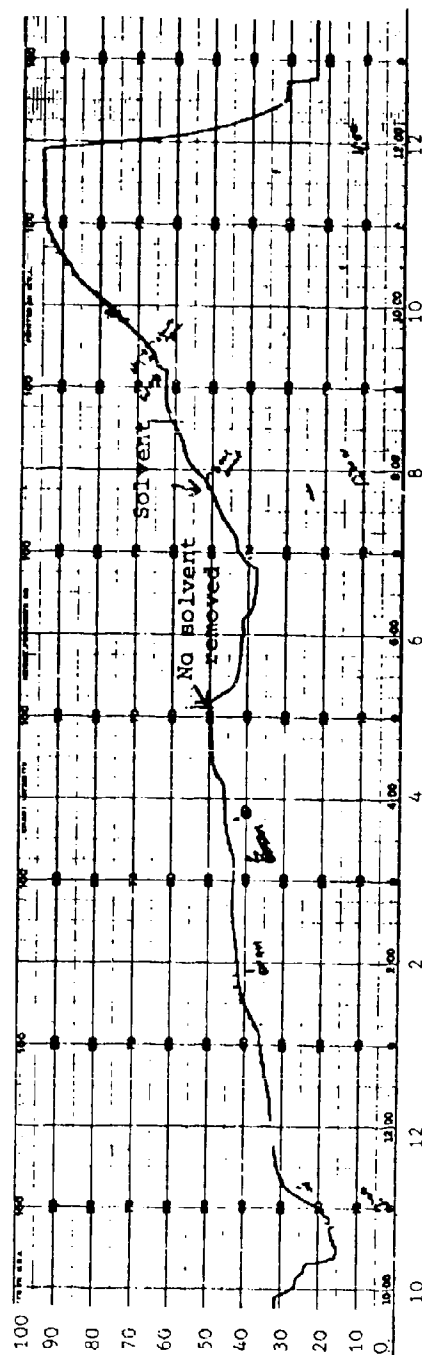


FIGURE C-12
Solvent strip for Extraction 7

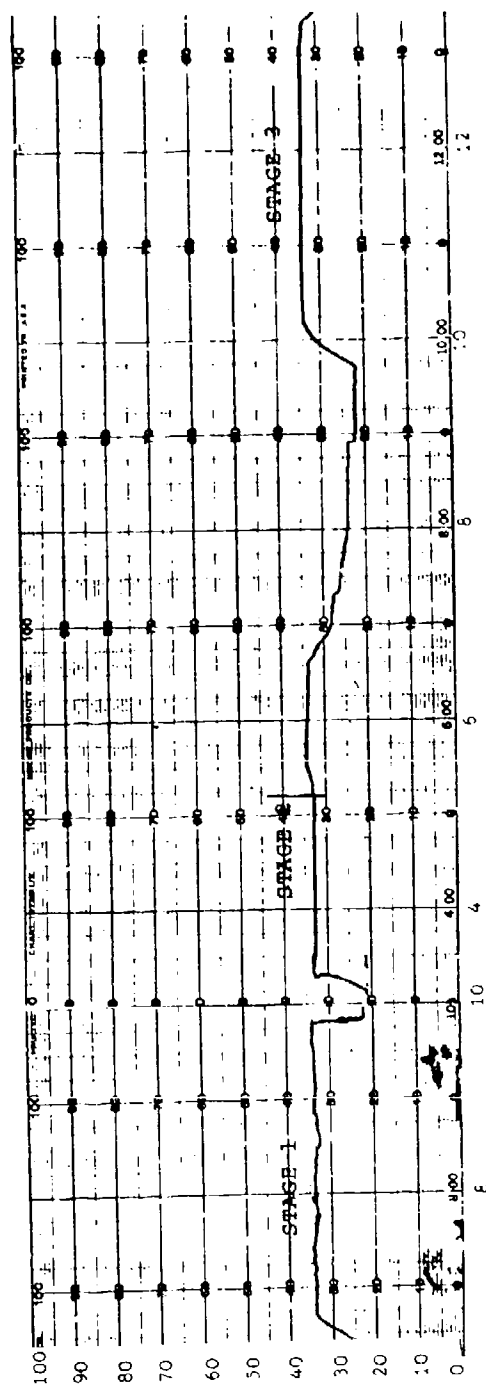


FIGURE C-13
Temperature trace for Extraction 8

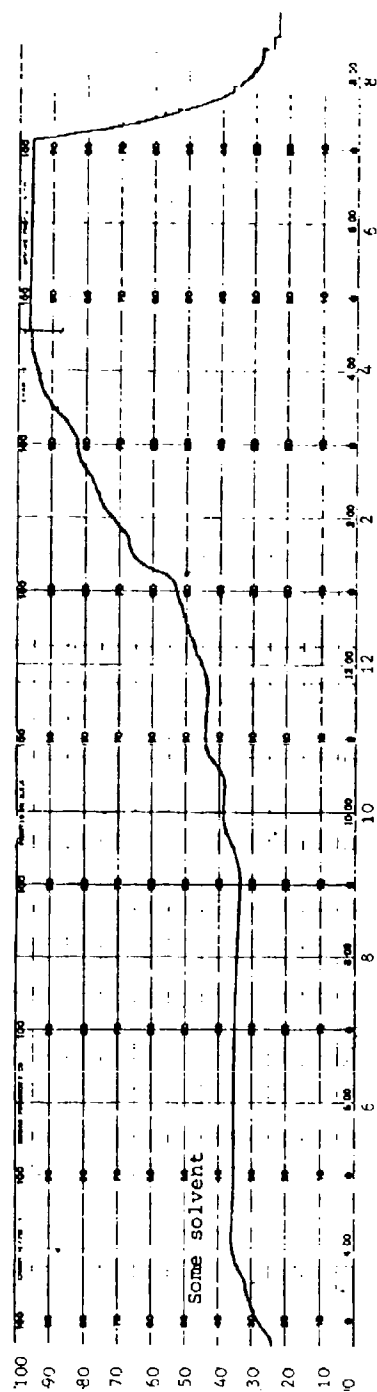


FIGURE C-14
Temperature trace for solvent strip - Extraction 8

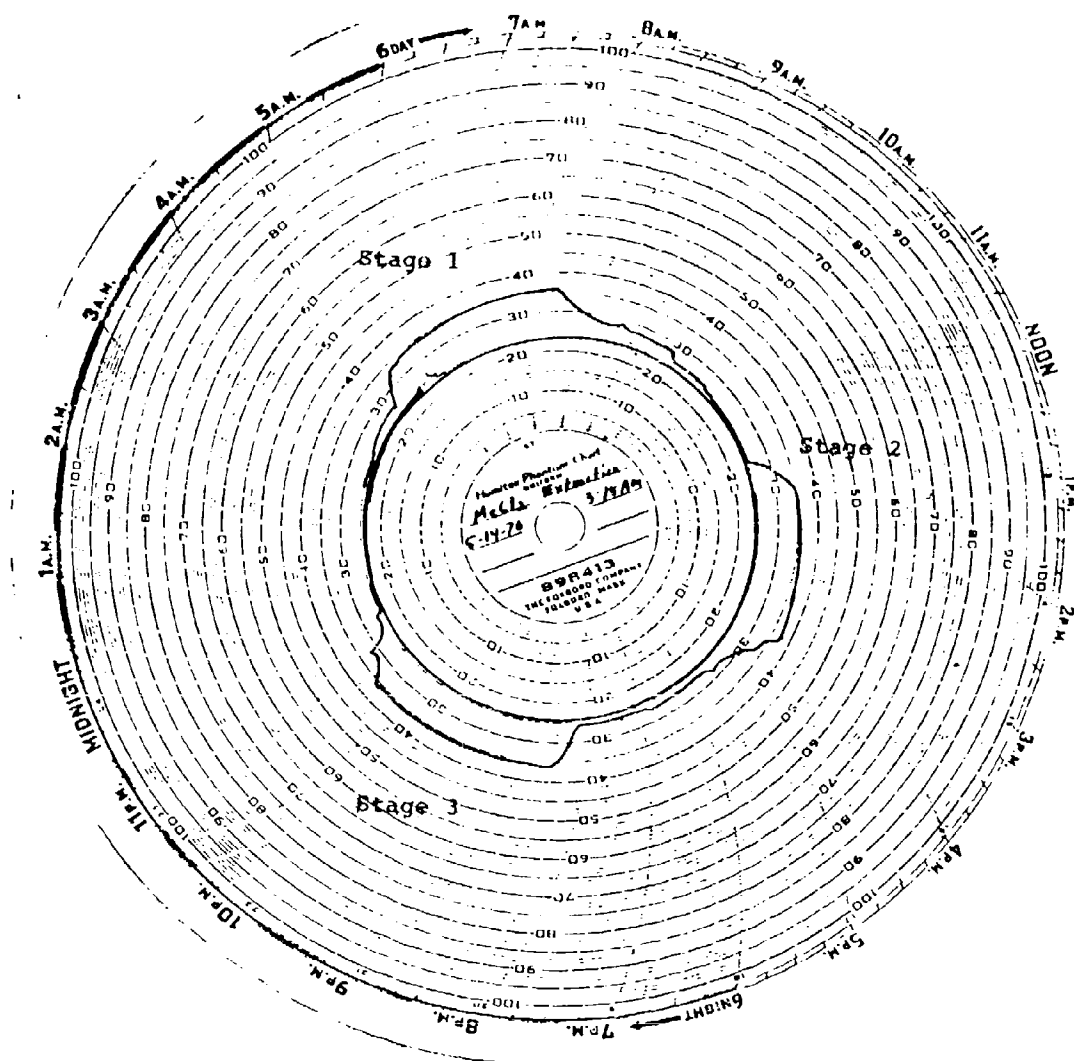


FIGURE C-15
Temperature trace for extraction 9

APPENDIX D

BALL POWDER PREPARATION

HARDENING

1. Work Plan
2. Standard Operating Procedure

WORK PLAN

A. Hardening and Shaping

1. Four hundred pound hardenings will be made using standard Pilot Plant operating procedures for the 1,000 gallon still.
2. Enough FNH will be hardened to produce 150 lbs. each of .034"/.028" and .028"/.020" for coatings.

STANDARD OPERATING PROCEDURE
HARDENING FOR ALL STILLs

Materials:

	<u>#2 & #3 Stills</u>	<u>#4 & #5 Stills</u>	<u>1,000 gallon</u>
Nitrocellulose	50 lbs.	5 lbs.	500 lbs.
Water	450 lbs.	60 lbs.	4,000 lbs.
Ethyl Acetate			
Ratio to NC			
for: .029"/.017"	3.4:1.0	Same	Same
.017"/fines	4.0:1.0	Same	Same
Diphenylamine	227 gms.	22.7 gms.	5 lbs.
Colloid	1.5 lbs.	227.0 gms.	15 lbs.
Na ₂ SO ₄ (dry)	12.5 lbs.	567.0 gms.	125 lbs.

Procedure:

Prior to starting of a hardening batch in the 100 gallon still, the following steps should be carried out:

1. Check the empty still to be certain that it is clean. If it is not clean, wash thoroughly with water and if necessary, run a clean out as per clean out SCP.
2. Drain all water from the still jackets. Be certain to close drain and open line from jacket to steam trap.
3. Add several inches of water to the still and check to see that the dump valve is tightly closed and not leaking.
4. Empty the condensor solvent receiver and salvage any usable ethyl acetate. Be certain that the vent line from solvent receiver is left open to the atmosphere after this job is completed.
5. Add water and nitrocellulose.
6. Start agitation at solution speed.

	RPM
#3 Still	150
#2 Stills	110
#4 & #5 Stills	60
1,000 Gallon Still	60

7. Add ethyl acetate and diphenylamine solution to still.
8. Check level of contents of the still and adjust level by addition of water.
9. Heat contents of still to 68°C. and hold for one hour.

10. At the end of solution period, adjust speed of agitator to RPM desired for graining step, stop agitation and allow to coalesce at 68°C. for:

5 minutes in 10 gallon still
10 minutes in 100 gallon still
15 minutes in 1,000 gallon still.

11. Check to make certain lacquer floats.

12. Toward the end of the solution period, take a sample of the lacquer and note its consistency. If material is too stiff on pulling apart, not enough solvent was used. If material is too rubbery, too much solvent was used.

13. Start agitation at speed indicated on the following schedule for graining:

Schedule:

	#2			#3			#4 and #5			1,000 gal still		
	Time	Temp.	RPM	Time	Temp.	RPM	Time	Temp.	RPM	Time	Temp	RPM
Solution	1 hr.	68°C	110	1 hr.	68°C	150	1 hr.	68°C.	60	1 hr.	68°C	60
Coalesce	10 min	68°C	0	10 min	68°C	0	5 min	68°C.	0	15min	68°C	0
Graining:	1 hr.	68°C		1 hr.	68°C		1 hr.	68°C.		1 hr.	68°C	
39/25			50			50			40			35
25/17			60			60			45			50
17/fines			80			80			50			60
Salt Addn.	1 hr.	68°C		1 hr.	68°C		1 hr.	68°C.		1 hr.	68°C	
Shaping &	2 hrs	68°C		2 hrs.	68°C		2 hrs	68°C.		2 hrs	68°C	
Dewatering												
Distillation:	2 hrs	First	GS	2 hrs.	First	GS	2 hrs	First	GS	2 hrs	First	GS
		Vapor			Vapor			Vapor			Vapor	
	2 hrs	75°C	GS+10									
	1.5 hrs	80°C	GS+20									
		85°C	GS+30		Same			Same			Same	
		90°C	GS+40									
		95°C	GS+50									
	1 hr.	99°C	GS+70									
		99°C	GS+70									

14. After coalesce period, dissolve colloid in hot water and add to still.

10 gallon still - 227 gms colloid in 800 ml. H₂O
100 gallon still - 1.5 lbs. colloid in 6 lbs. H₂O
1,000 gallon still - 15 lbs. colloid in 60 lbs. H₂O

15. Continue graining period for one hour.

16. At the end of the one hour graining period, add salt to contents of the still over a one hour period. Still level at this point should just permit washing of still contents over skimmer paddle.
17. Continue to shape and dewater for an additional two hours at 68°C. to desired RPM. Sample and observe the batch from time to time and make notes in notebook regarding size and shape.
18. When shaping and dewatering is completed, start heating the batch for distillation using two hours time to raise the temperature from 68°C. to the temperature where the first vapor starts coming off.

(Note: Be sure cooling water is flowing through the condensor and that there is sufficient room in the solvent receiver for the material distilled from the still.)
19. When the first vapor starts coming off, continue with distillation according to the schedule outline.
20. When the temperature of the batch reaches 99°C., continue distilling at 99°C. for 1/2 hour, then cool, and wash with three cold water washes to remove salt and colloid.
21. Drain still contents into barrels for wet screening.
22. Turn off cooling H₂O.

APPENDIX E

BALL POWDER PREPARATION COATING

1. Work Plan
2. Operating Instructions:
 - NG Impregnation
 - Deterrent Coating

WORK PLAN

A. Coatings -

1. Three, fifty pound NG and deterrent coatings, for each cut, will be made using standard operating procedures to make WC 870 and WC 846 propellant.
2. One hundred pounds each of WC 870 and WC 846 propellant will be shipped to BAAP for testing.

OPERATING INSTRUCTIONS
NG IMPREGNATION - 100 GAL. STILL

1. Check still for cleanliness - Run clean out if necessary.
2. Add enough water to still to check dump valve for proper seating.
3. Empty solvent receiver to make space for condensed solvent.
4. Write up request in proper book, (D or N Log Book)
5. Weigh out requested amount of powder using powder pyc, and dump into still. (Pyc. factor .391)
6. Fill still from back side of manhole with water as per Attachment 4.
7. Turn on agitator and adjust to 140 RPM and add colloid as requested.
8. Heat to 50°C. and hold for 30 min.
9. Weigh out NG-EA as per % requested for powder following NG Handling S.O.P.
10. Add NG-EA to still over 1 1/2 hours (max. time) as per NG Handling S.O.P.
11. After NG is in, raise temperature to 65°C. over one hour time and hold for four hours unless otherwise requested.
12. Sample liquor for Q.C. to analyze for NG.
13. Start vacuum distillation and bring to 11.5 inches over 20 min. period to avoid surging.
14. Raise temperature 2°C. per hour till 86°C. is reached or as 15 below.
15. Start R.S. samples for Q.C. at 76°C. and sample each hour till Q.C. cuts distillation.
16. After Q.C. cuts distillation, cool and process as requested.
17. Make sure all information is recorded in proper log book.

OPERATING INSTRUCTIONS-NG IMPREGNATION-100 GAL STILL (Continued)

	<u>Powder</u>	<u>H₂O and Pdr. Based on H₂O Level</u>		<u>Water Level</u>	<u>lbs./inch</u>	<u>Colloid Temp</u>		<u>NG/EA Add</u>
#2 - 100 gal.	100 lbs.	450 lbs.		25"	25 lbs/in.	2 lbs.	50°C	2 hours
#3 - 100 gal.	100 lbs.	450 lbs.		29"	25 lbs/in.	2 lbs.	50°C	2 hours
#4 & #5 - 10 gal.	15 lbs.	60 lbs.		16"	5 lbs/in.	100 gms	50°C	30 min.

	<u>RPM</u>	<u>NG Impregnator.</u>		<u>Vacuum Distillation</u>	<u>Temperature Rise</u>	<u>Hold</u>
		<u>52-65°C.</u>	<u>Hold 65°C.</u>			
#2 - 100 gal.	140	1 hr.	3 hrs.	11 1/2"	2°C./Hr. 65-86°C.	86°C.-2 hr.
#3 - 100 gal.	140	1 hr.	3 hrs.	11 1/2"	2°C./Hr. 65-86°C.	86°C.-2 hr.
#4 & #5 - 10 gal.	140	1 hr.	3 hrs.	11 1/2"	2°C./Hr. 65-86°C.	86°C.-2 hr.

OPERATING INSTRUCTIONS

DETERRENT COATINGS

	<u>100 Gallon Still</u>	<u>10 Gallon Still</u>
Water	450 lbs.	60 lbs.
Colloid	900 grams	150 grams
Wt. of Powder (max.)	125 lbs.	20 lbs.
Deterrent, %	As Requested	As requested
Temp.: E.C.	86°C.	86°C.
DBP	76°C.	76°C.
Agitator RPM's	140	140

1. Heat still to temperature as per table above.
2. Add deterrent emulsion as per table below unless otherwise requested.

Deterrent Emulsion

	<u>100 Gallon Still</u>	<u>10 Gallon Still</u>
Water	20 lbs.	5 lbs.
Colloid	2 lbs.	5 grams
Deterrent, %	As Requested	As Requested
Add to Still	1 hour	1/2 hour

3. Heat treat as per request.
4. Record all data such as pick up, powder bonding, or anything unusual in proper "D" book.
5. Cool still by turning off the steam valve and opening the water valve to the jacket of the still.
6. Wash powder and dump.
7. Turn off all cooling water to still jacket and condenser.

Operating Instructions - Deterrent Coatings

8. Label and process as per request.
9. Clean up and hose down area.

APPENDIX F

FINISHED POWDERS

TABLE F-1	Powders for WC 870 Blending
F-2	Propellant Description Sheet X3492 (WC 870 Speed)
F-3	Ballistic Acceptance Test X3492 (WC 870 Speed)
F-4	Powders for WC 846 Blending
F-5	Propellant Description Sheet X3491 (WC 846 Speed)
F-6	Ballistic Acceptance Test X3491 (WC 846 Speed)

TABLE F-1
POWDERS FOR WC 870 BLENDING

	Base Stock	Final Powder	Base Stock	Final Powder	Base Stock	Final Powder
Identification	AG-3154	AB20693-5	AG-3156	AB20706-8	AG-3156	AB20717-10
Coating No.		N 9219		N 9237		N 9821
Qty. of Pdr.	66.5	73	65	73	25	27
Granulation (in)	.034/.028	-	.034/.028	-	.034/.028	-
Ave. Grain Dia. (in)	0.0318	-	0.0299	-	0.0299	-
Spec. Gravity	1.51	-	1.518	-	1.518	-
Grav. Dens. gm/cc	0.931	0.942	0.918	0.900	0.918	0.901
Diphenylamine, %	-	-	1.0	-	1.0	-
Dinitrotoluene %	-	-	0.17	-	0.17	-
Chlorine, %	0	-	0	-	0	-
Dibutylphtha- late, %		5.20	0.1	6.36	-	6.15
Nitroglycerine %	-	9.53	-	4.98	-	9.05
Potassium Ni- trate, %		0.66		0.97		0.54
Tin Dioxide, %		0.84		1.00		0.68
M & V, %		0.97		0.82		1.01

TABLE F-2

CO FORM 15 Apr 52 1201	ORDNANCE CORPS PROPELLANT DESCRIPTION SHEET	Supersedes CO Form 1204 dated 1 Jul 49 which is obsolete																																																																																	
U.S. Army Lot No. <u>X3492</u>	Composition No. <u>AB20732-3</u>	For <u>20mm</u> <u>Badger</u>																																																																																	
Manufactured at <u>Olin Corp., St. Marks, FL</u>	Packed Weight <u>14 lbs.</u>																																																																																		
Contract No. _____ Date _____	Specification No. <u>MIL-P3984</u>	Revision of <u>Apr. 13, 1969</u>																																																																																	
Accepted blends (Nos.) _____ Blend of rework material _____ Drawing <u>C10534813</u> dated <u>9/30/71</u>																																																																																			
Nitrogen Content	K. I. Starch Test (65% C)	Stability Test (135° C)																																																																																	
Maximum _____	Maximum _____ Mins.	Maximum _____ Mins.																																																																																	
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GRAIN DIMENSIONS	FINISHED GRAIN (INCHES)																																																																																		
	Manufacturer	Inspector																																																																																	
Length (L) _____	Opening _____	% Retained _____																																																																																	
Diameter (D) _____	U.S. # 18 _____	0.20 _____																																																																																	
Diameter of perforations (d) _____	20 _____	16.80 _____																																																																																	
Web {	Inner _____	25 _____	55.51 _____																																																																																
	Outer _____	30 _____	27.02 _____																																																																																
	Average _____	35 _____	0.50 _____																																																																																
	Calculated _____	40 _____	0.06 _____																																																																																
Difference between inner and outer web in per cent of web average	Pay _____	0.86 _____																																																																																	
L:D (Y) _____	Total _____	100.15 _____																																																																																	
D:d (X) _____																																																																																			
Date packed <u>11/8/76</u> Date offered <u>11/8/76</u> Date sampled <u>11/8/76</u> Date test finished <u>11/10/76</u> Date description sheets forwarded _____ Type of Packing Box <u>Fiber Pak</u> Remarks: _____																																																																																			
Superintendent _____	Inspector of Ordnance _____	U.S. Chemist _____																																																																																	

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TABLE F-3

OS-602 Rev. 1

TABLE F-4
POWDERS FOR WC 846 BLENDING

Identification	N-9202		N-9214		N-9243		N-9243		N-10054		N-10054	
	Base Stock AG 3155	Final Powder	Base Stock AG 3155	Final Powder	Base Stock	Final Powder	Base Stock	Final Powder	Base Stock	Final Powder	Base Stock	Final Powder
Coating No.	AG 3155 & 51 AB20685-6	111.36	AG 3155 & 51 AB20693-6	AB20712-9	AG 3156 AB20721-5	AG 3156 AB20717-2	AG 3156 AB20721-4					
Qty. of Powder	100	28.5	97	50	3	-	50	63	3	-	70	
Gratulation	28/20	-	28/20	23/20	-	28/20	-	28/20	-	28/20	-	
Ave. Grain Dia.	0.0215	-	0.0215	0.0242	-	-	-	0.0242	-	-	-	
Spec. Gravity	1.563	-	1.563	1.538	-	-	-	1.538	-	-	-	
Grav. Dens. gm/cc	0.916	0.926	0.916	0.891	0.952	0.891	-	0.891	0.911	0.891	-	
Diphenylamine, %	0.71	-	0.71	0.87	-	-	0.938	0.87	-	-	-	
Chloride, %	0	-	0	0	-	0	-	0	-	0	-	
Dibutylphthalate	-	3.91	-	-	5.65	-	-	-	6.22	-	-	
M & V	-	0.84	-	-	-	-	-	-	-	-	-	
Nitroglycerine	-	9.21	-	-	8.99	-	6.99	-	7.42	-	7.42	
Web (inches)	-	0.0147	-	-	0.0137	-	0.0128	-	0.0112	-	0.0168	

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TABLE F-5

CO FORM 15 Apr 52 1204		ORDNANCE CORPS PROPELLANT DESCRIPTION SHEET		Supersedes CO Form 1204 dated 1 Jul 49 which is obsolete	
U.S. Army Lot No. <u>X3491</u>		Composition No. <u>AB20736-4</u>		For <u>7.62mm.</u>	
Manufactured at <u>Olin Corp., St. Marks, Fla.</u>		Specification No. <u>MIL-P3984E</u>		Packed Weight <u>34 lbs.</u>	
Contract No. _____ Date _____		Revision of <u>Apr. 13, 1969</u>		Drawing <u>C10534784</u> dated <u>9/27/71</u>	
accepted blend (Nos) _____ Blend of rework material					
Nitrogen Content		K I Starch Test (65.5° C)		Stability Test (150° C)	
Maximum _____	_____	Maximum _____	_____	Maximum _____	_____
Minimum _____	_____	Minimum _____	_____	Minimum _____	_____
Average _____	_____	Average _____	_____	Average _____	_____
				Explosion _____	_____
MANUFACTURE OF PROPELLANT					
Total weight of solvent per pound NC _____ Consisting of _____ pounds alcohol and _____					
pounds other per 100 pound solvent. Percentage of remix to whole _____					
PROCESS SOLVENT RECOVERY AND DRYING					
TEMP. °C				TIME	
From _____	_____			Days _____	Hours _____
The following analyses are an average of two tests					
TESTS OF FINISHED PROPELLANT					
COMPOSITION			STABILITY AND PHYSICAL TESTS		
Constituent	Formula	Mt.	Insp.	Mt.	Insp.
Nitroglycerin		8.65		120° C heat test, S.P.	70 min.
Dinitrotoluene		0.11		Explosor	300 min.
Diphenylamine		0.81		Dust & Foreign Material	0.04
Diethylphthalate		4.98		Graphite	0.10
Methylene Chloride Extr.		4.55			
Nitrocellulose		85.17		Grav. density, or pounds per cu. ft.	9.38
Total Volatiles		1.13		Tin Dioxide	.0165
Moisture & Volatiles		0.75		Web	
Residual Solvent		0.73		Nitrogen	13.12
Calcium Carbonate		0.09			
Sodium Sulfate		0.09			
GRAIN DIMENSIONS			FINISHED GRAIN (INCHES)		
			MEAN VARIATION IN PERCENT OF MEAN DIMENSION		
			Manufacturer	Inspector	
Length (L) _____ Granulation			Opening	% Retained	
Diameter (D) _____			U.S. # 20	2.41	
Diameter of perforations (d) _____			25	9.02	
Inner _____			30	41.32	
Outer _____			35	39.87	
Web { Average _____			40	6.21	
Calculated _____			45	0.90	
Difference between inner and outer web			Pan	0.35	
in per cent of web average _____			Total	100.08	
L:D (Y) _____					
D:d (X) _____					
Date packed <u>11/19/76</u> Date offered <u>11/19/76</u> Date sampled <u>11/19/76</u>					
Date test finished <u>11/21/76</u> Date description sheets forwarded _____					
Type of Packing Box <u>Fiber Pak</u>					
Remarks: _____					
Superintendent		Inspector of Ordnance		U.S. Chemist	

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BALLISTIC ACCEPTANCE TEST FOR SMALL ARMS PROPELLANT POWDER										Lot No. <u>X-3491</u> Caliber <u>7.62</u> Type <u>BALL</u> Date <u>11/21/76</u>			
TESTED IN ACCORDANCE WITH SPECIFICATION													
PROPELLANT POWDER				GUNS				Proof Fired By _____					
M: LIN MATHIESON CHEMICAL CORPORATION								Head Blend # _____					
Lot No. <u>X-3491</u>				Receiver No. _____				Bullet Type <u>M-80 BALL</u>					
Made at <u>St. Marks, Fla.</u>				Barrel No. _____				Bullet Wt. <u>147</u> Gr.					
Net Wt. of Lot <u>34</u> Lbs.				Barrel Length <u>22"</u>				Primer <u>LC</u>					
Charge <u>47.0</u> Gr.				Head Space <u>1.631</u>				Cartridge Case <u>LC</u>					
Air Space <u>+0.047</u> Ins.				Times Fired _____				Temperature <u>70</u> °F					
DATE FIRED		BLEND TEST		GUN OR GAGE NUMBER		NO. OF RDS.		CORRECTED MEAN VELOCITY AT 78 FT.		CORRECTED PRESSURE		VARIATION IN VELOCITY AND CHAMBER PRESS.	
11/21/76		Blend of <u>6</u>		4		20		2762		MEAN MAX.		EXTR. STD. DEV.	
		Samples <u>4</u>		4		20		47600 49800		6200 1464			
ACTION TIME (MILLISECONDS)		TEMPERATURE TESTS				CHANGES				PORT PRESSURE TEST			
AVE.	MAX. IND.	NO. OF RDS.	STORED AT	FIRED AT	MAX. IND. PRESS.	VEL.	PRESS.	PORT PRESS.	NO. RDS.	ACTUAL AVE.	CORR. AVE.		
1.33	1.38	20	-65°F	-65°F	56000	-37	+4700		20	10578	11178		
		20	+125°F	+125°F	48000	-7	+100						
REMARKS Chamber Pressure +3 SD= 51,992 psi													
DATE FIRED	LOG NUMBER			CHARGE GRAINS	GUN OR GAGE NUMBER	NO. OF RDS.	MEAN VELOCITY AT 78 FT.		STANDARD REFERENCE CARTRIDGE				
11/21/76	Standardization				4	20	2741		LC-Y-7.62-903				
	Standard Firing						2714		Mean Pressure				
	Correction						+27		Chamber Port				
11/21/76	Standardization				4	20			41,700 12,900				
	Standard Firing								40,500 12,300				
	Correction						+1200		+600				
THIS LOT PROPELLANT POWDER _____ THE BALLISTIC TEST REQUIREMENTS.													
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